Metrology Efforts at the APS

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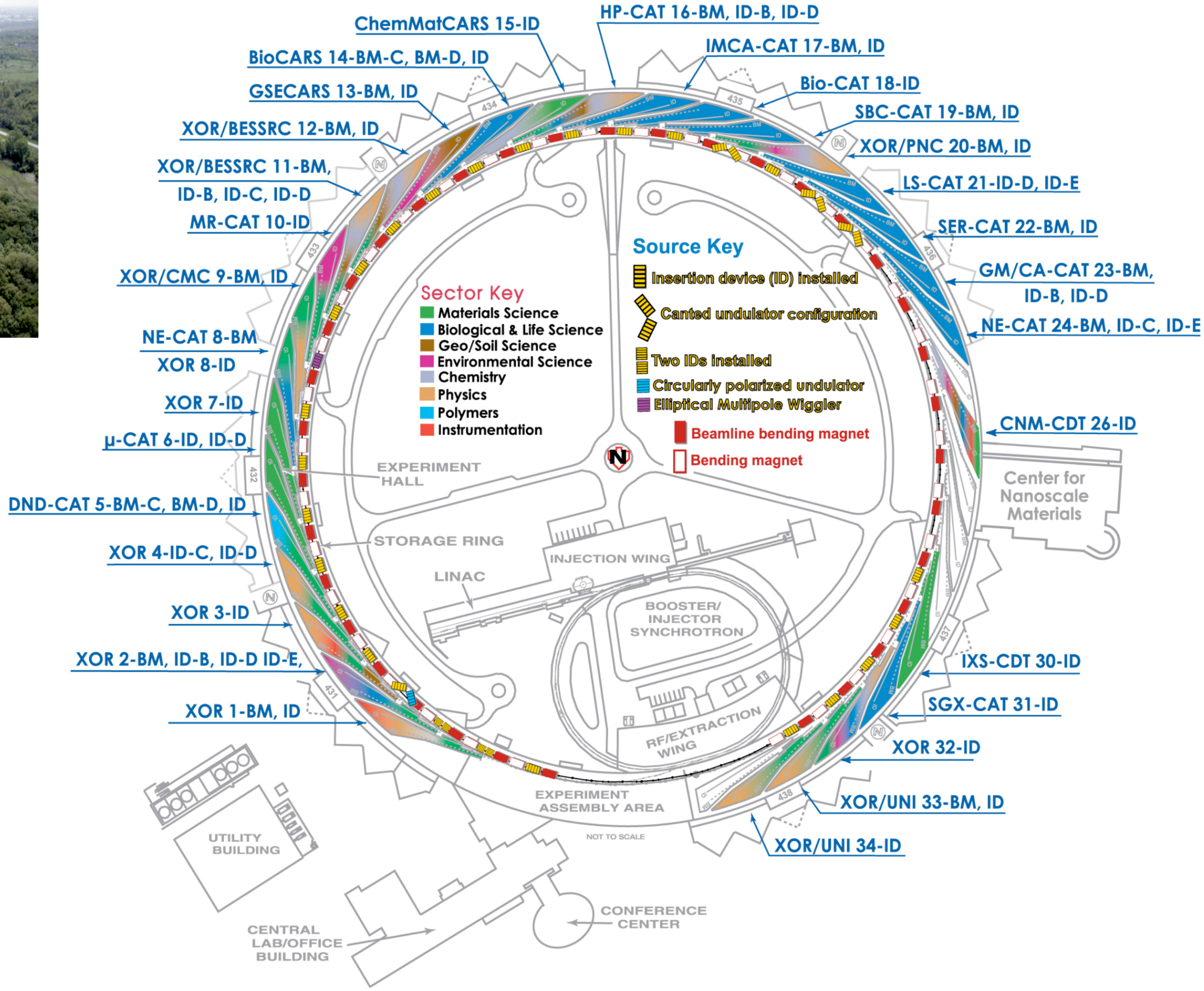
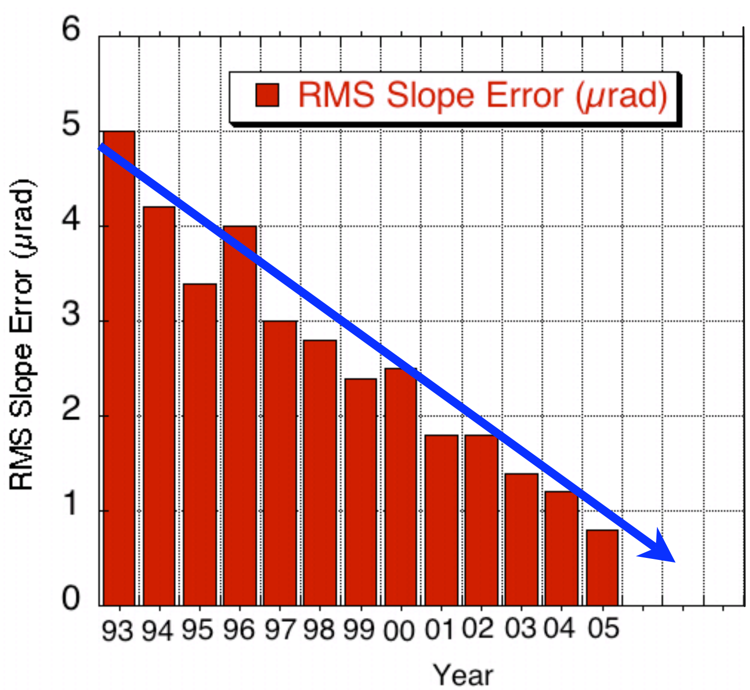
The Advanced Photon Source at Argonne

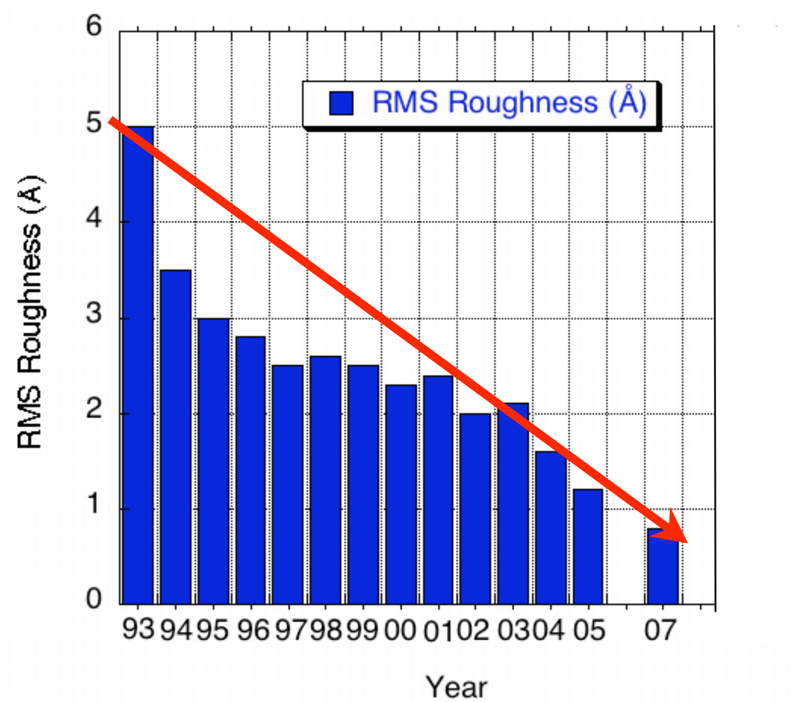


Outline

- Introduction
- Optical metrology efforts
- At-wavelength metrology developments
- Summary

Evolution of surface quality of large hard x-ray mirrors during 1993-2007



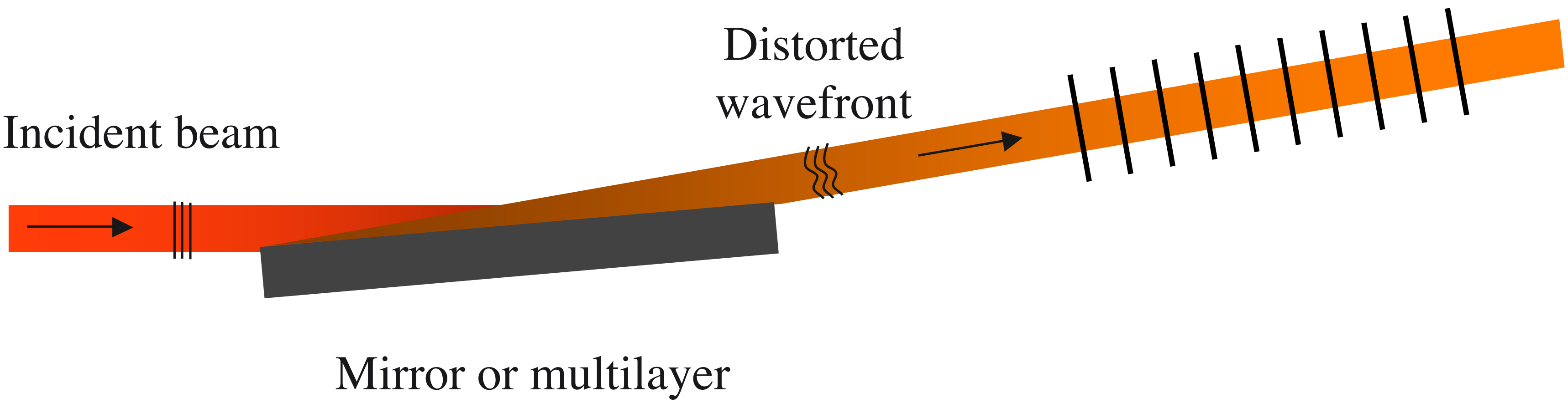


- Compiled from APS, ESRF, and SPring-8 data
- Mirror materials: Si, ULE Glass, Fused Silica, Zerodur (1 m or larger)
- Mirrors acquired from various vendors

Future mirror quality requirements and metrology

- Future synchrotron radiation sources such as NSLS-II, as well as X-ray FEL and ERL sources, will provide beams with ultra-low emittance and high brightness.
- Improving optics surface quality will be crucial to many experiments including those using micro- and nanofocusing, inelastic and small angle scattering, and phase imaging and experiments requiring coherence preservation.
- Mirrors with slope errors (rms, standard deviation) less than a fourth of the source divergence interecepted by a mirror will be needed.
- This means that mirrors with surface slope error in the nanoradian range and roughness <1 Å rms will be required. This is well beyond the quality of currently commercially available mirrors that are typically 0.5 to 1 µrad rms for flat mirrors and higher for curved mirrors.
- Moreover, one must be able to characterize the surface quality with metrology tools whose accuracy are at least equal to, but ideally three times smaller than, the specified figure error. This presents a challenge to existing metrology tools.
- This beyond the capability of existing optical metrology instruments, and at-wavelength metrology tools and techniques may be critical for developing, characterizing, and optimizing next-generation beamline optics and components.

Future mirror quality requirements: coherence-preserving mirrors



- Propagation of a coherent beam over distance D with a phase perturbation φ filtering with maximum contrast spatial period $p_M = 1/f$

$$\tilde{I}(f) = \delta(f) + 2R(f)\ \gamma(\lambda D f)\ \sin(\pi\lambda D f^2)\ \tilde{\varphi}(f)$$ (J.P. Guigay)

$$p_M = \frac{\sqrt{2\lambda D}}{\sin\theta}$$

D=0.5 m to 5 m p_m =2.6 to 8 mm at 3 mrd incidence 20 kev

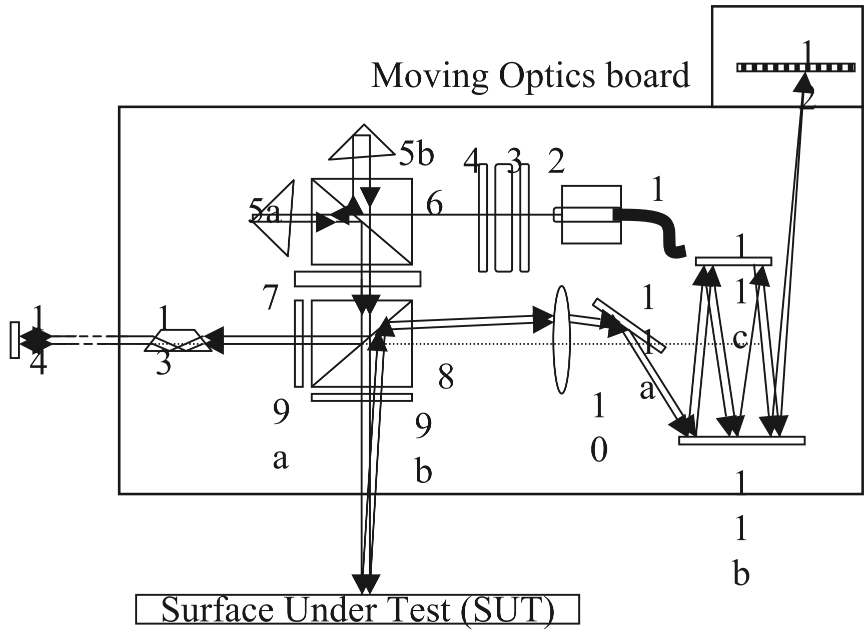
$$\varphi = \frac{4\pi z \sin\theta}{\lambda}$$

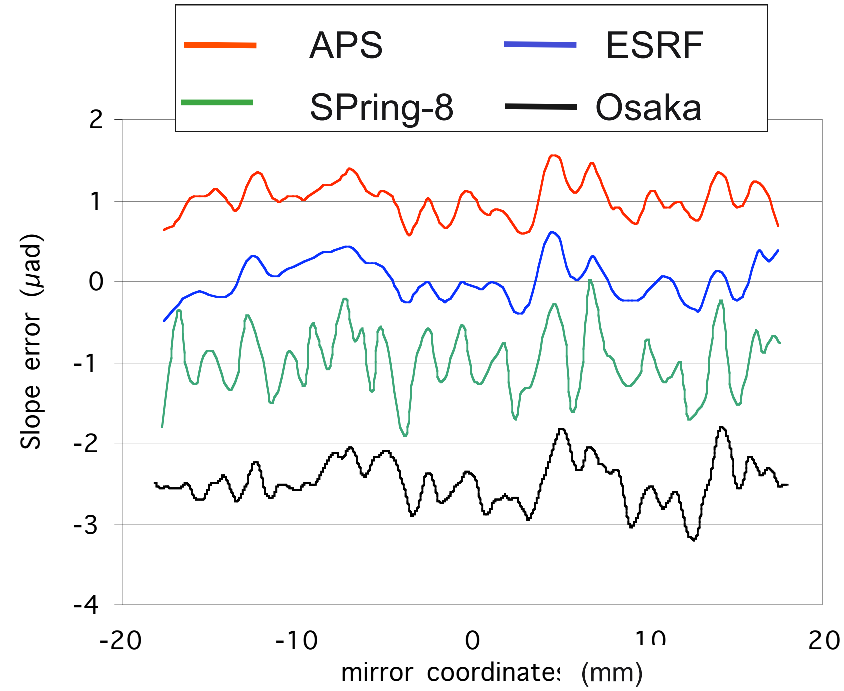
Contrast of 20% → 1.8 Å figure error tolerance (O. Hignette)

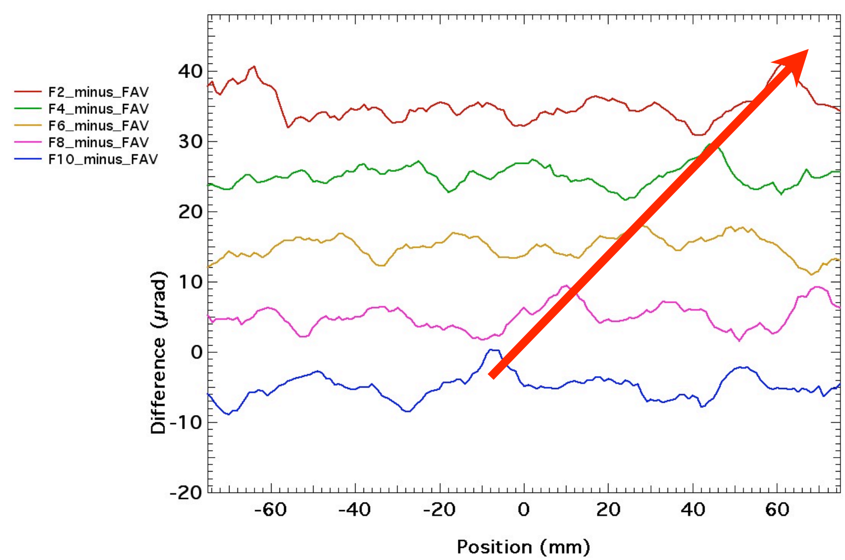
Optical metrology efforts at the APS

APS/ESRF/SPring-8 the long trace profiler (LTP) round robin

APS Argonne NATIONAL LABORATORY · ESRF · SPring-8 · Osaka University





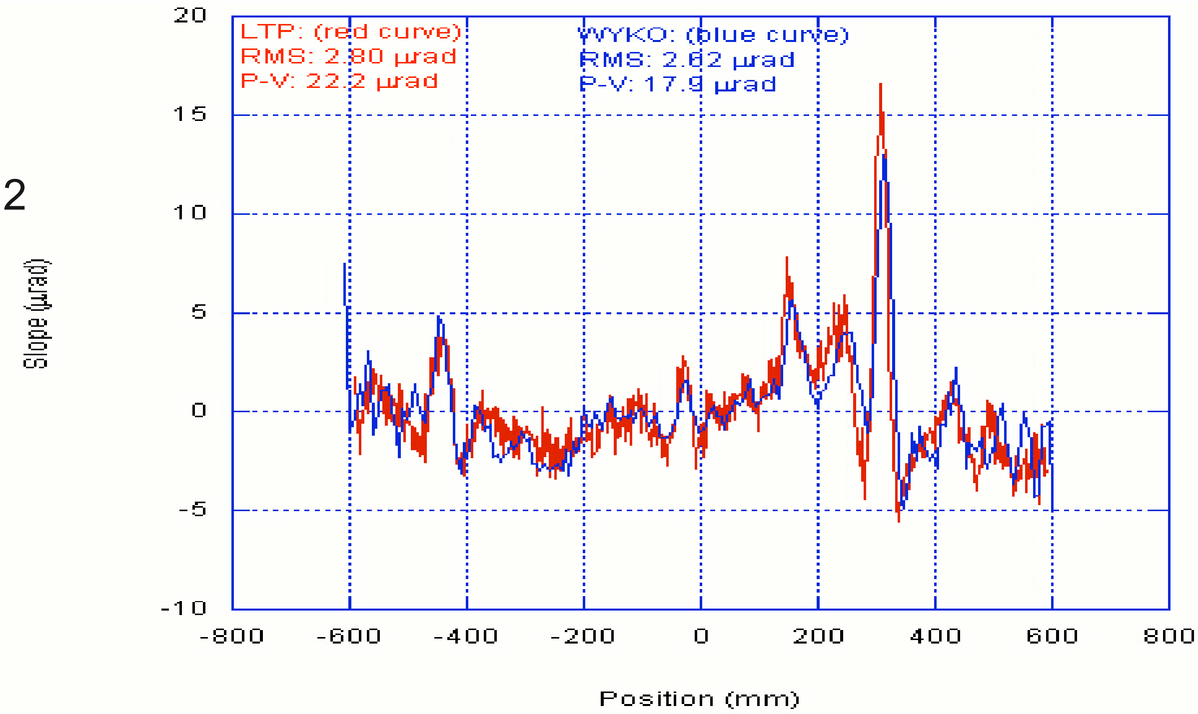


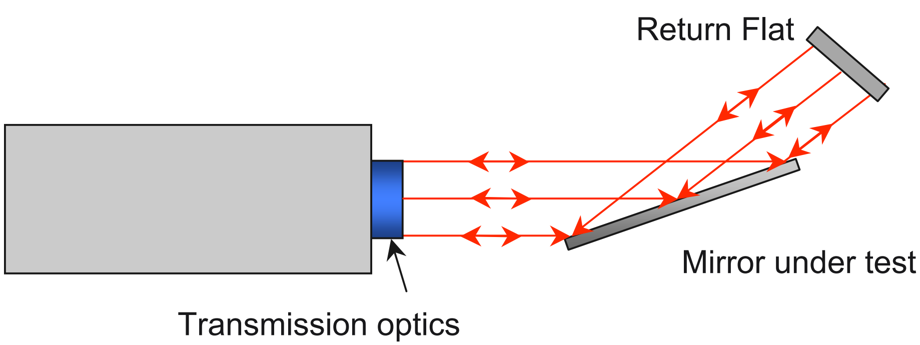
- Mean radius of curvature matching < 0.3%
- Slope error rms agreement < 0.2 μrad
- Shape error rms agreement < 0.2 nm

Wyko-6000 laser interferometer upgrades - Comparison with the APS LTP II

New Wyko-6000

- CCD camera 1024x1024 pixels, 12 bit digitation
- New acquisition and analysis software & computer (Intelliwave Corp.)
- Spatial resolution:
  - 146 µm @ normal incidence
  - ~ 1 mm for 1 m long mirror
- Stitching capability

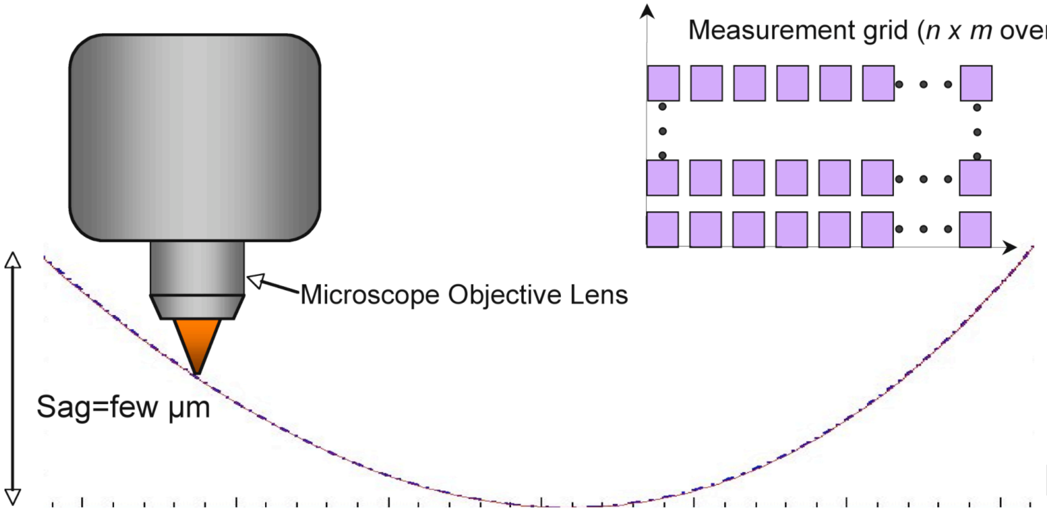


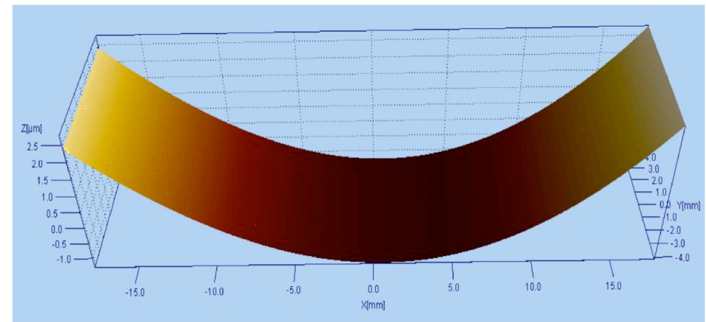


J. Qian, L. Assoufid, and A. Macrander, SPIE **6057** (2007)

- Advantage over the LTP: Area measurement instead of a line profile for LTP
- Disadvantage: requires a null optic for evaluation of highly curved optics.
- Stitching can overcome this limitation.

Development of microstitching interferometry and metrology of precisely-figured K-B mirrors





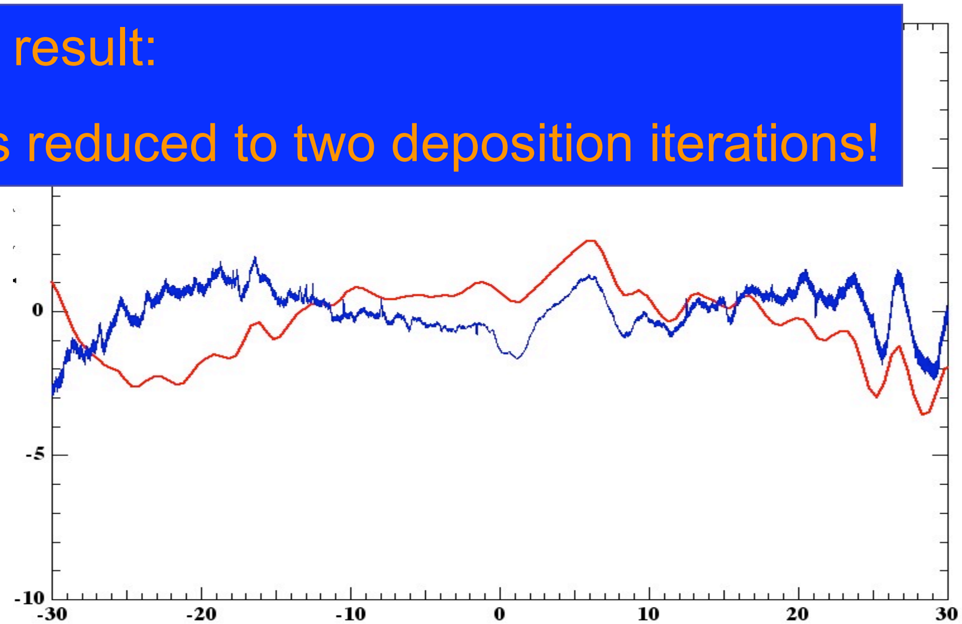
Profile of a profile-coated Pt KB Mirror

The result:

Profile coating of elliptical thin films reduced to two deposition iterations!

Best fit parameters:

- LTP data
  - S2 = 125.2 mm
  - Theta = 2.74 mrad
- Stitched data
  - S2 = 125.6 mm
  - Theta = 2.72 mrad



L. Assoufid *et al.* "A Microstitching Interferometer for Evaluating the Surface Profile of Precisely Figured Hard X-ray K-B Mirrors," SPIE Proc. Vol 6704 (2007)

Ongoing and future R&D

- Optical metrology tools to meet next-generation optics requirements
  - Develop a slope measuring device with nanoradian resolution
  - Further improve our stitching capability

At-wavelength/x-ray in-situ metrology

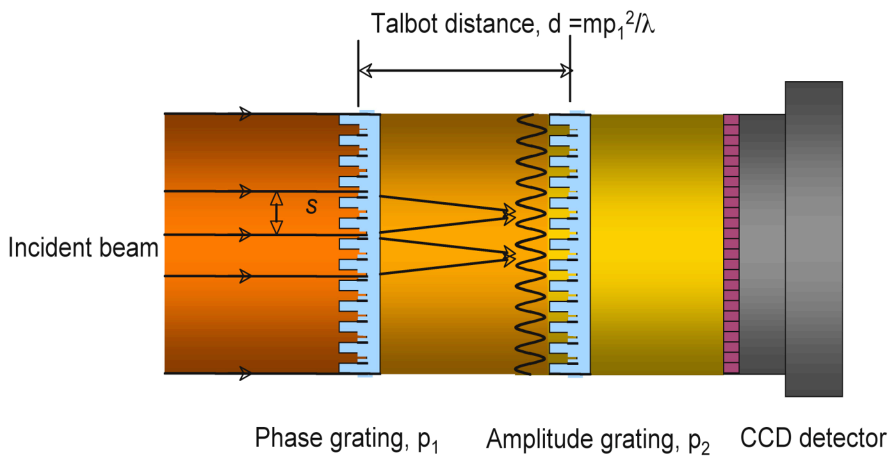
Why at-wavelength metrology is essential

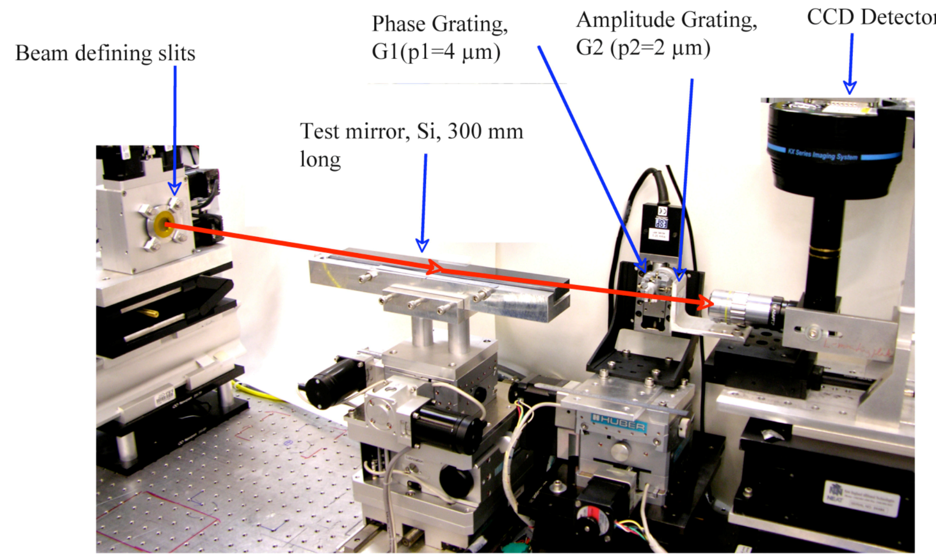
- The small emittance of third-generation x-ray synchrotron radiation sources provides a means to acquire surface metrology data more accurately than currently available optical metrology tools.
- Existing optical metrology tools are blind to possible material nonhomogeneity of the optics reflecting surface.
- For multilayers the surface information obtained is directly related to the actual reflecting planes, which might differ from the top surface probed by the visible light metrology.
- With x-rays, turbulence effects are minimal because the air refractive index is close to one.
- At-wavelength metrology may be essential for:
  - characterization of coherence-preserving optics
  - calibrating optical metrology tools
  - developing in-situ beam wavefront monitoring tools
  - validating optics performance simulation codes
- Disadvantages:
  - Requires precious beamtime, can be time consuming and costly, not practical for optics manufacturers.

At-wavelength metrology methods

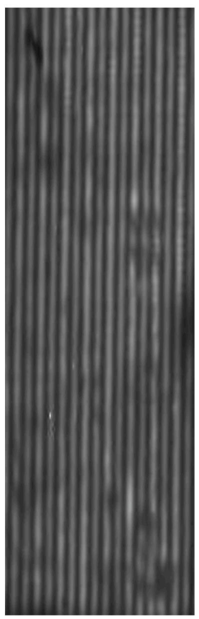
- X-ray LTP with a sensor based on a pencil beam sequential Hartmann (O. Hignette @ ESRF)
- Interferometry: grating/Talbot interferometer (ESRF, APS),
- Speckle inteferometry
- Phase retrieval (Osaka University/SPring-8)
- Inverse propagation (A. Souvorov, ESRF/SPring-8)
- Reflectivity, diffuse scattering

Development of at-wavelength metrology: x-ray grating interferometer

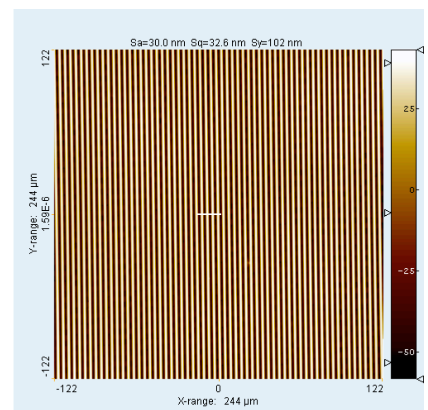




Test setup at 8-ID beamline (L. Assoufid, C. Rau, M. Sprung, A. Sandy) (April 2007, unpublished)

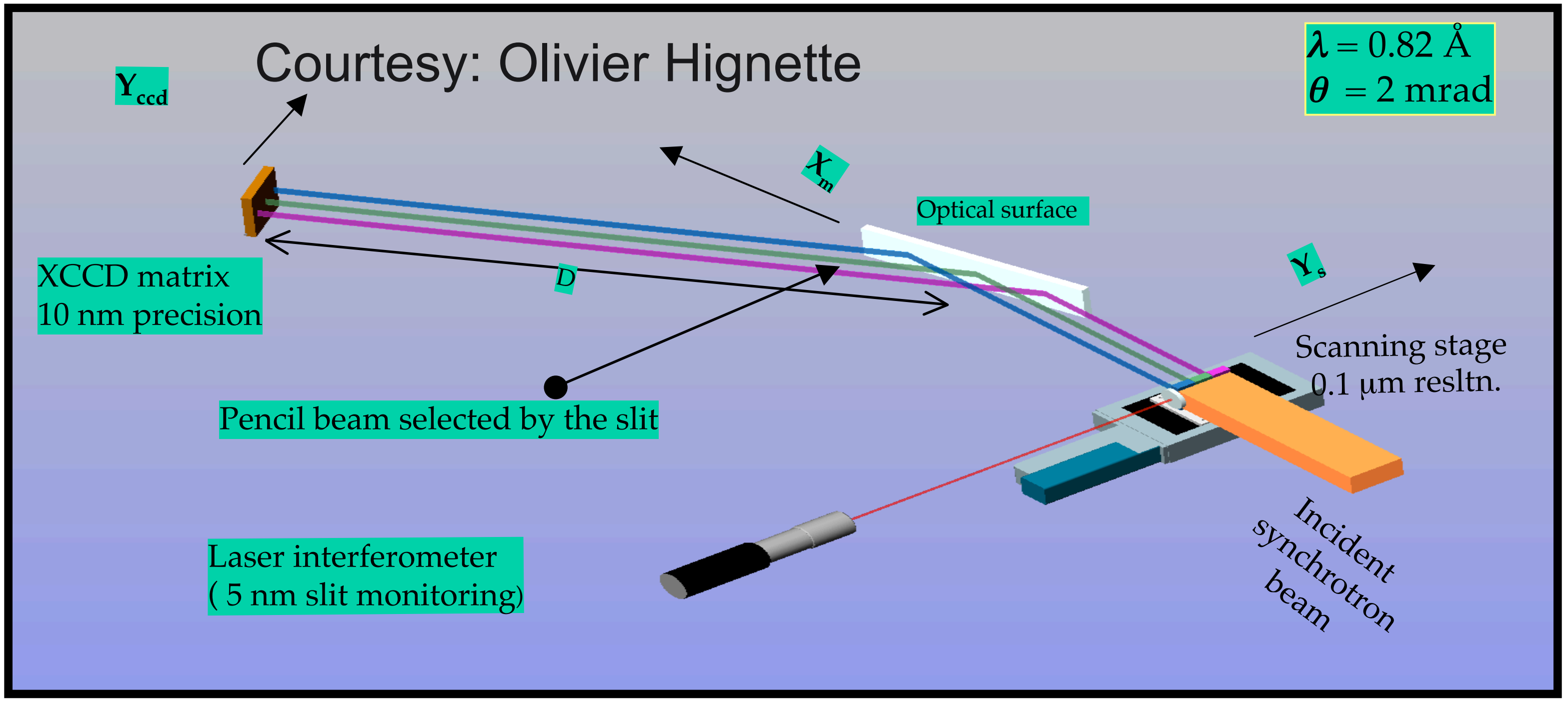


- Fabrication of gratings is underway at CNM (by R. Divan, CNM) using deep-dry etching



At-wavelength in-situ metrology: X-ray LTP with a pencil beam sequential Hartmann sensor

- Case 1: Set-up for non-focused wavefront configuration

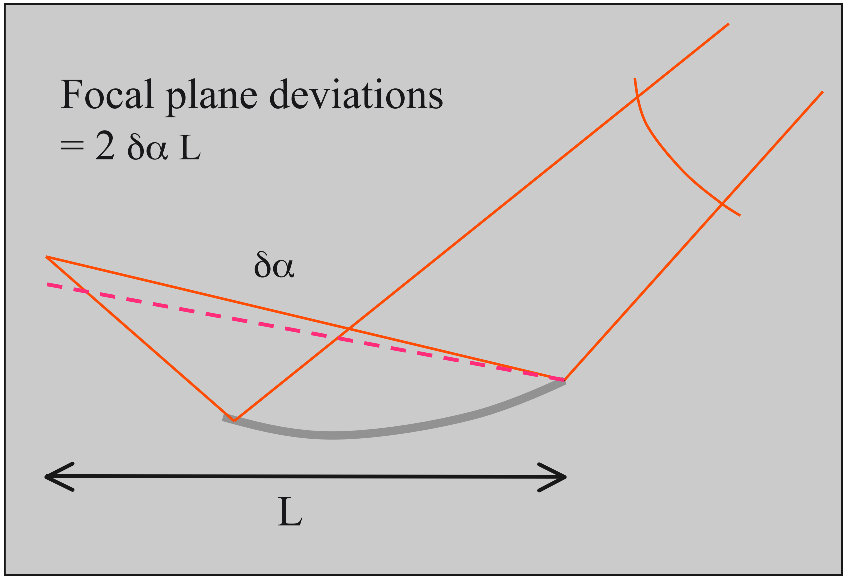


$$Mirror\ \ slope(X_m) = \frac{+Y_{CCD} + Y_S}{2(D - X_m)} - \left[\frac{-Y_{CCD} + Y_S}{2(D)}\right]_{REF}$$

(Courtesy: O. Hignette, ESRF)

At-wavelength in-situ metrology: X-ray LTP with a pencil beam sequential Hartmann sensor

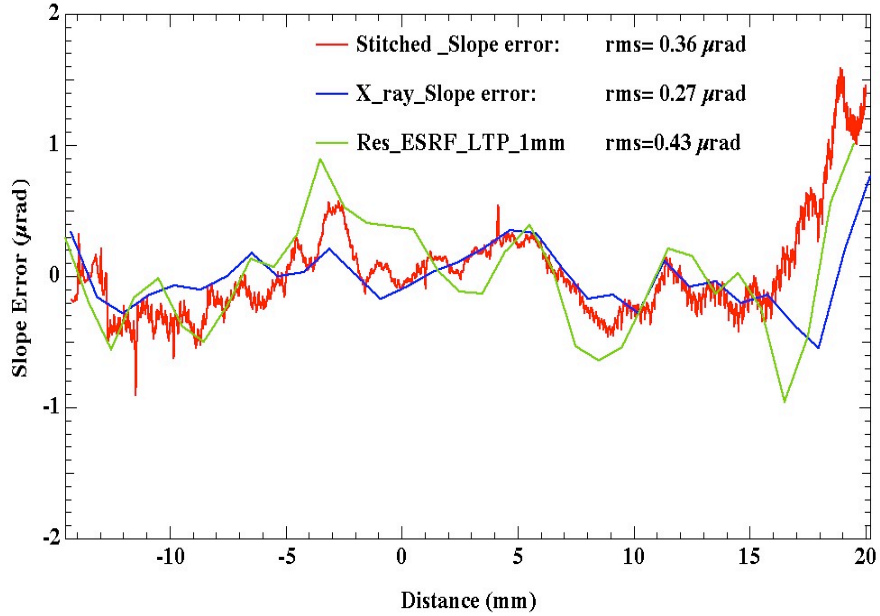
- Case 1: Set-up for focused wavefront configuration
  - The position-sensitive device (camera) is located very close to the focus.
  - The error with respect to the reference spherical wavefront centered on the focus plane is directly obtained.
  - The local slope error is given by:

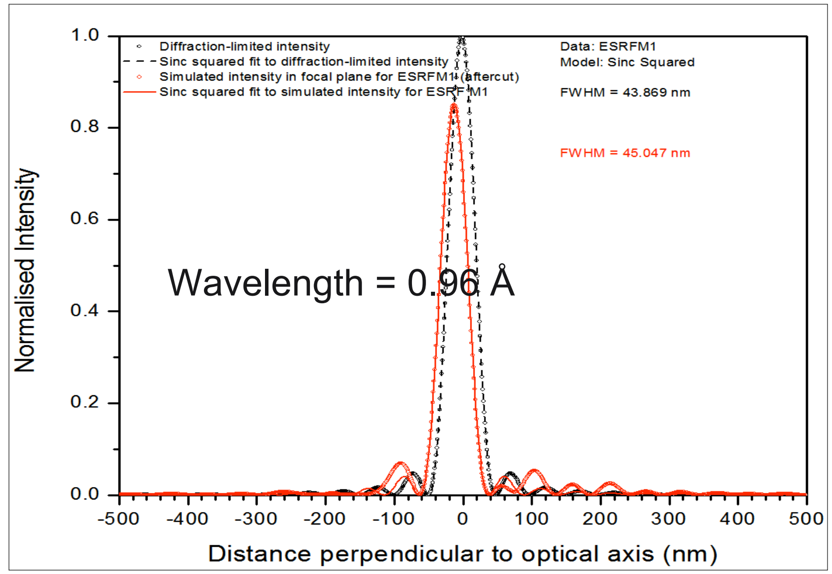


Courtesy: Olivier Hignette

$$Mirror\ slope\ error(X_m) = \frac{Y_{CCD}}{2D}$$

Evaluation of an APS profile-coated elliptical mirror for hard x-ray nanofocusing, with in-situ x-ray pencil beam profilometry





X-ray measurements done at ESRF ID-13 (98 m long) beamline (O. Hignette L. Assoufid, M. Burghammer, K. M. Kewish, C. Riekel—unpublished)

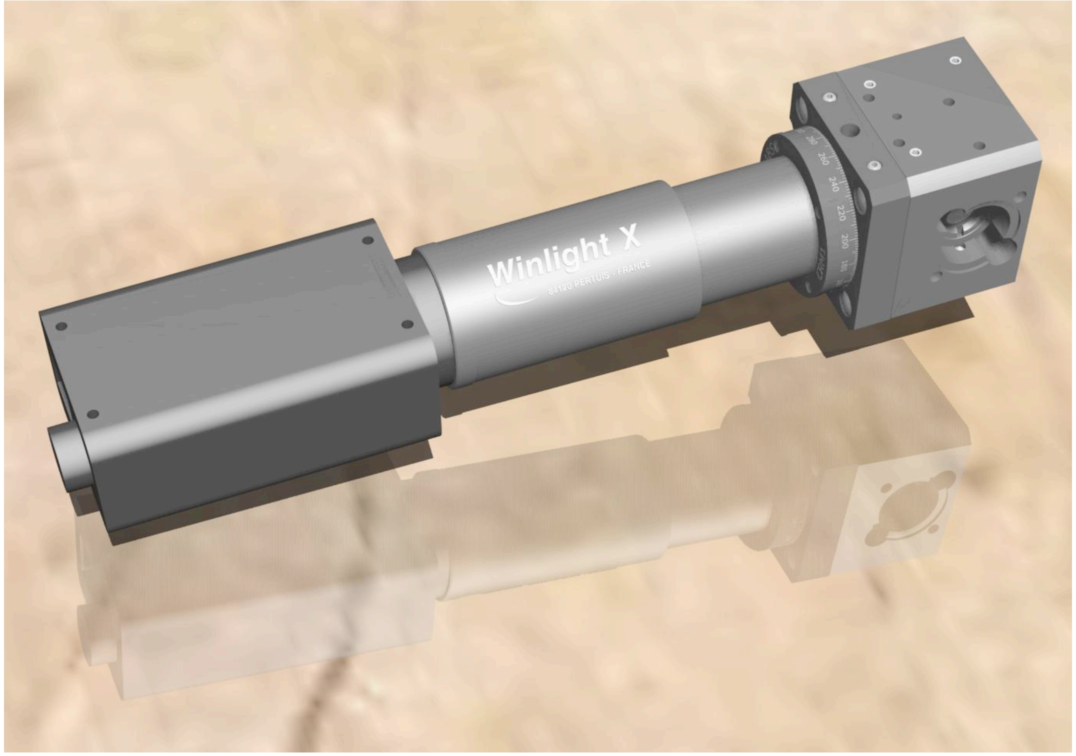
LTP Data: ESRF (by A. Rommeveaux, x-ray optics group)

Microstitched data: APS

Parameter	Design Value	Metrology data fitting	
		LTP (ESRF)	Stitching (APS)
Distance to source, s1 (m)	98	98	98
Distance to focus, s2 (mm)	77.5	74.3	76.4
Incidence angle (mrad)	3.9	3.58	3.67

Nanofocusing position sensor

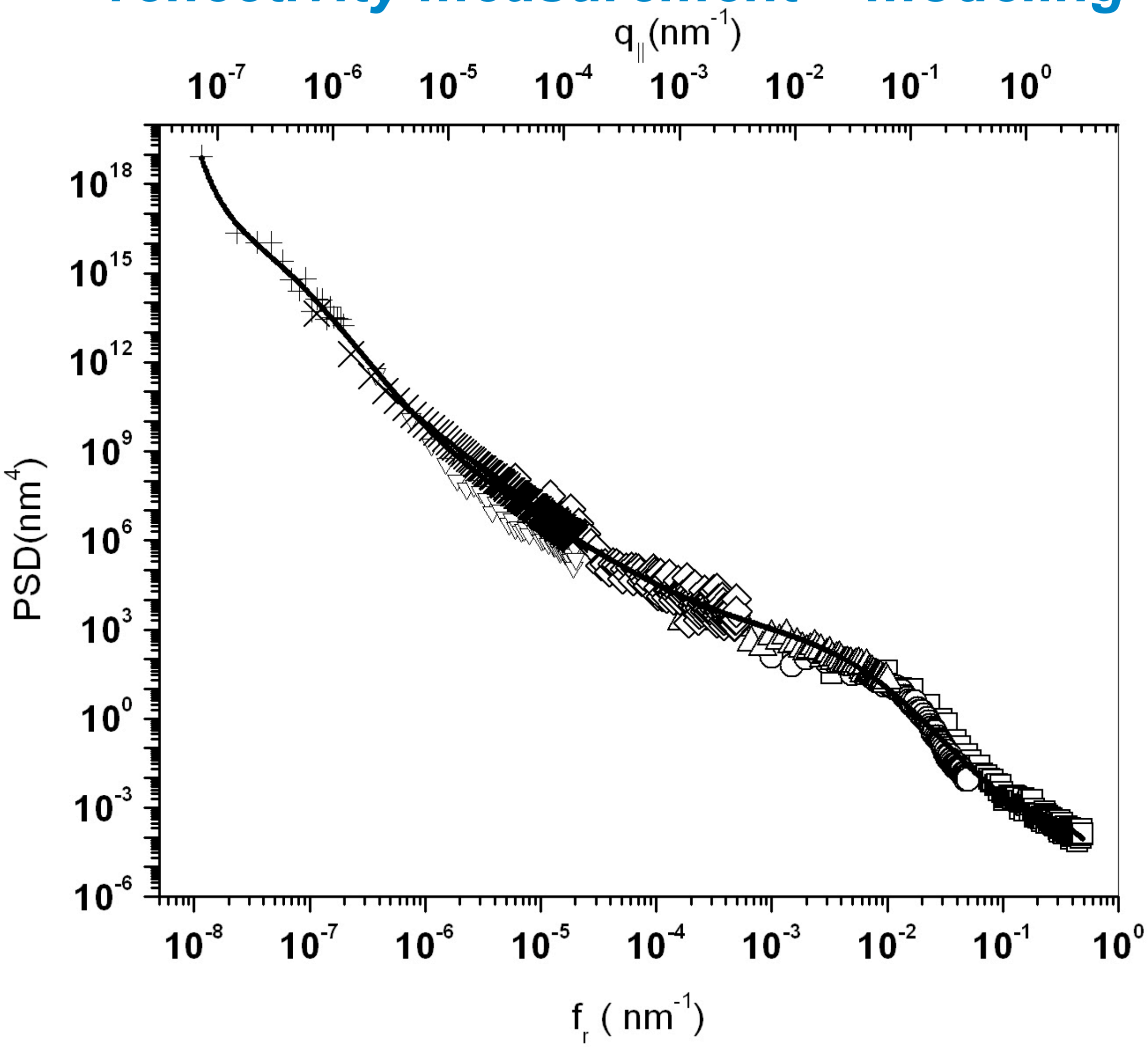
Expected position sensitivity: less than 2 nanometers rms

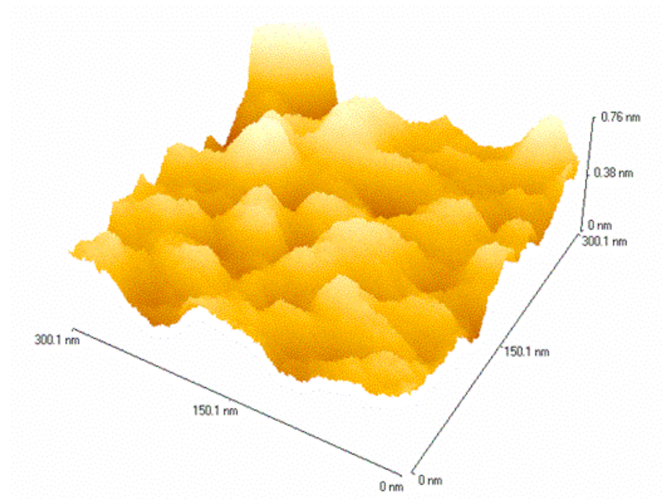


Courtesy: O. Hignette, WinlightX, France

High resolution XCCD camera (1034X779 pixels)
YAG:Ce thin luminescent screen
Field of view 800X600 μm
Pixel size 0.77 μm
Point spread function 2.3 μm FWHM
Measurement rate 30 HZ
GigaEthernet digital camera

Roughness evaluation from PSD - comparison with reflectivity measurement + modeling

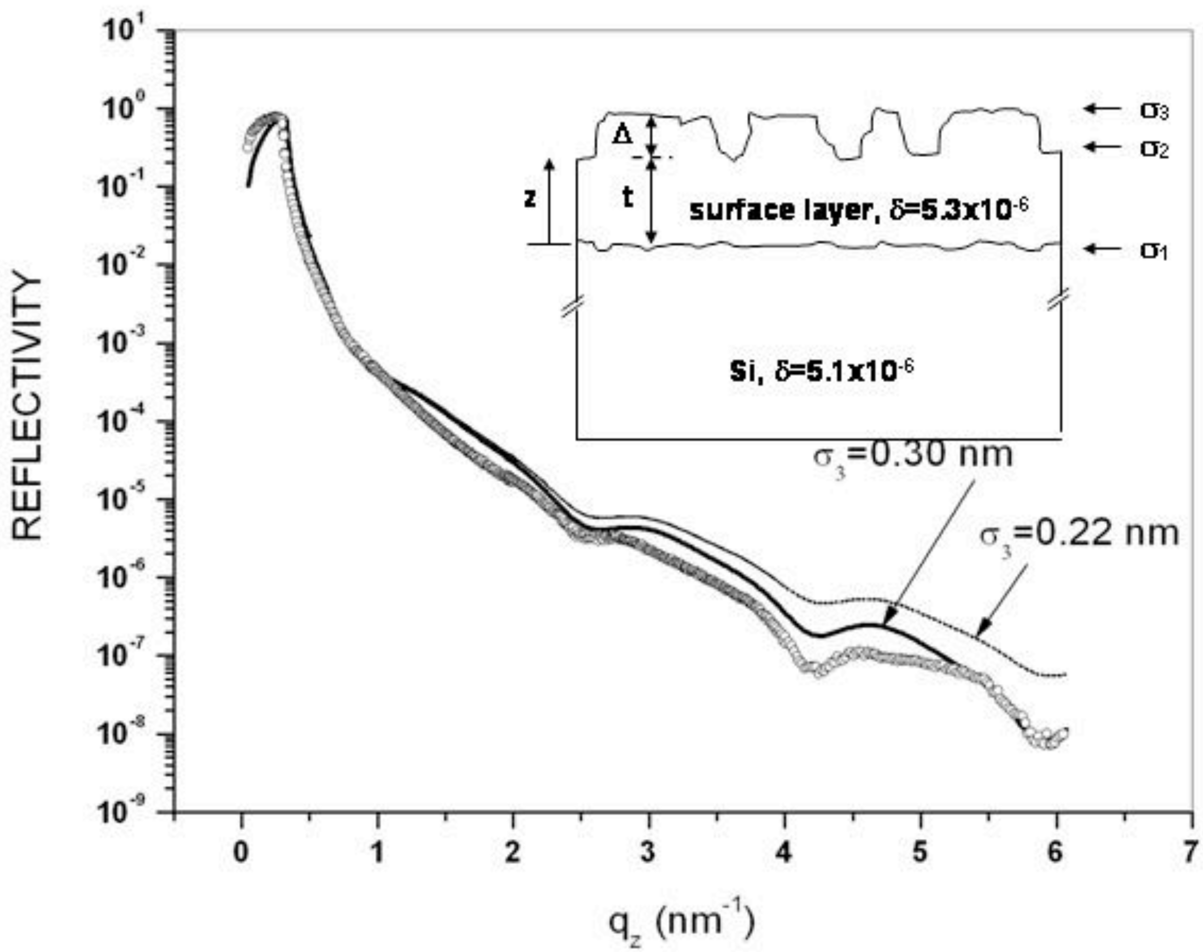




Power spectral density data and polynomial fit. LTP (+), TOPO 5x (∇), TOPO 15x (X), TOPO 40x (◊), AFM 6 μm (Δ), AFM 2 μm (O), AFM 0.3μm (�).

Roughness derived from reflectivity measurement and simulation

- The Optic: Si, Chemical-mechanically polished at the APS
- Measurements: APS 1-BM, @ E=10 keV



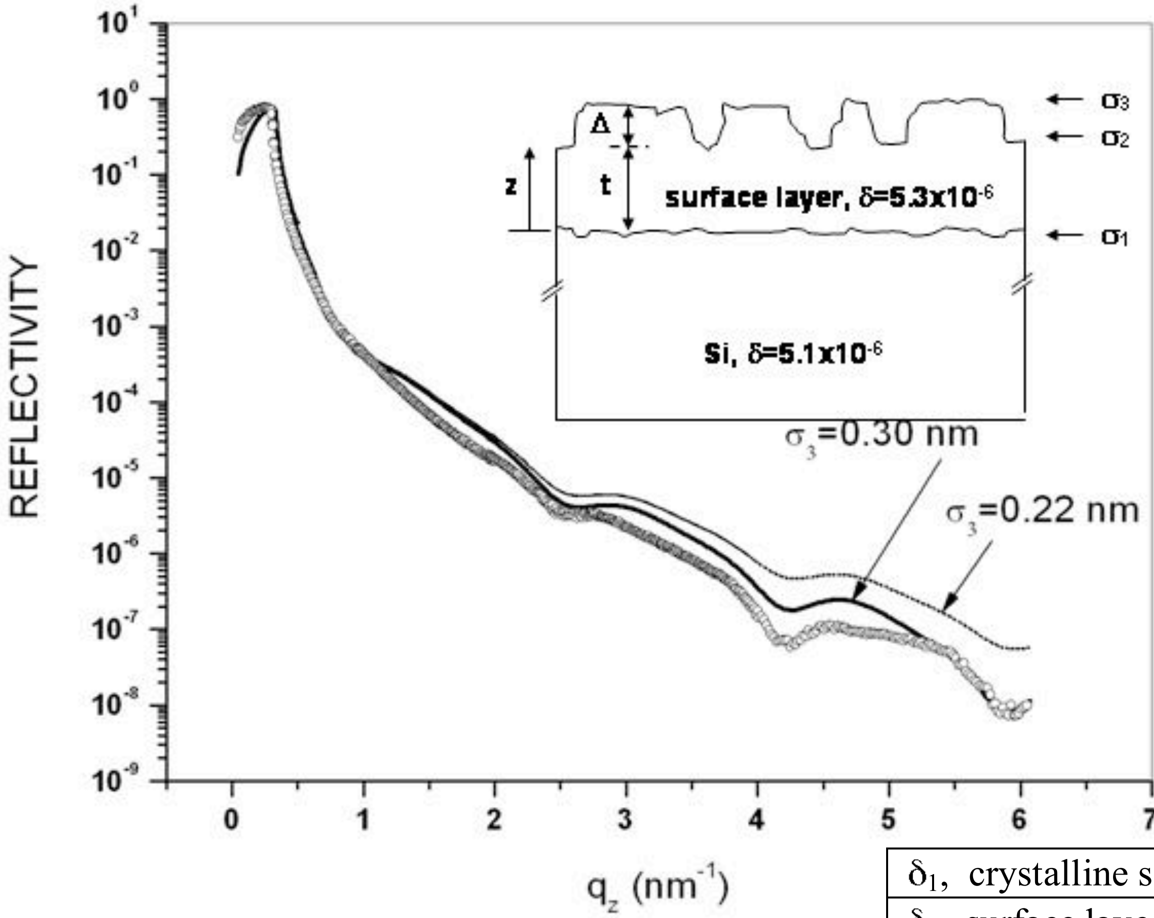
Reflectivity measurement and simulation

$$\frac{d\sigma}{d\Omega} = \frac{n_2^2}{q_z^2}\left| \iint dxdy e^{-iq_z z_2(x,y)} e^{-i(q_x x + q_y)} + \frac{(n_1 - n_2)}{n_2} \iint dxdy e^{-iq_z z_1(x,y)} e^{-i(q_x x + q_y)} \right|^2$$

[Ref: S. K. Sinha, E.B. Sirota, S. Garoff, H.B. Stanley, Phys. Rev. B, 38, 2297 (1988).]

$$\left.\frac{dq}{d\Omega}\right|_{spec} = \frac{4\pi^2 n_2^2}{q_z^2} A\delta(q_x)\delta(q_y) \left[\begin{array}{l} \left(\phi e^{-q_z^2\sigma_3^2/2} + (1-\phi)e^{-q_z^2\sigma_2^2/2}\right)^2 + \left(\frac{n_1 - n_2}{n_2}\right)^2 e^{-q_z^2\sigma_1^2/2} + \\ 2\left(\frac{n_1 - n_2}{n_2}\right)\left(\phi \cos(q_z(t+\Delta))e^{-q_z^2(\sigma_1^2+\sigma_3^2)/2} \quad + \quad (1-\phi)\cos(q_z t)e^{-q_z^2(\sigma_1^2+\sigma_2^2)/2}\right) \\ -4\phi(1-\phi)\sin^2(q_z\Delta/2)e^{-q_z^2(\sigma_2^2+\sigma_3^2)/2} \end{array} \right]$$

$$R = \frac{16\pi^2 n_2^2}{q_z^4} F \quad = \quad R_f F$$



$$R = \frac{16\pi^2 n_2^2}{q_z^4} F \quad = \quad R_f F$$

Parameters for the calculations

δ_1, crystalline silicon	5.1 x10^{-6}
δ_2 , surface layer	5.3 x10^{-6}
thickness, t	3.60 nm
land height, Δ	3.80 nm
land coverage fraction , ϕ	0.85
roughness of substrate/layer interface, σ_1	0.22 nm
roughness of layer top surface, σ_2	0.22 nm
roughness of tops of lands, σ_3	0.22 nm, 0.30 nm

X-ray Reflectivity and Power Spectral Density of Smoothly Polished Silicon

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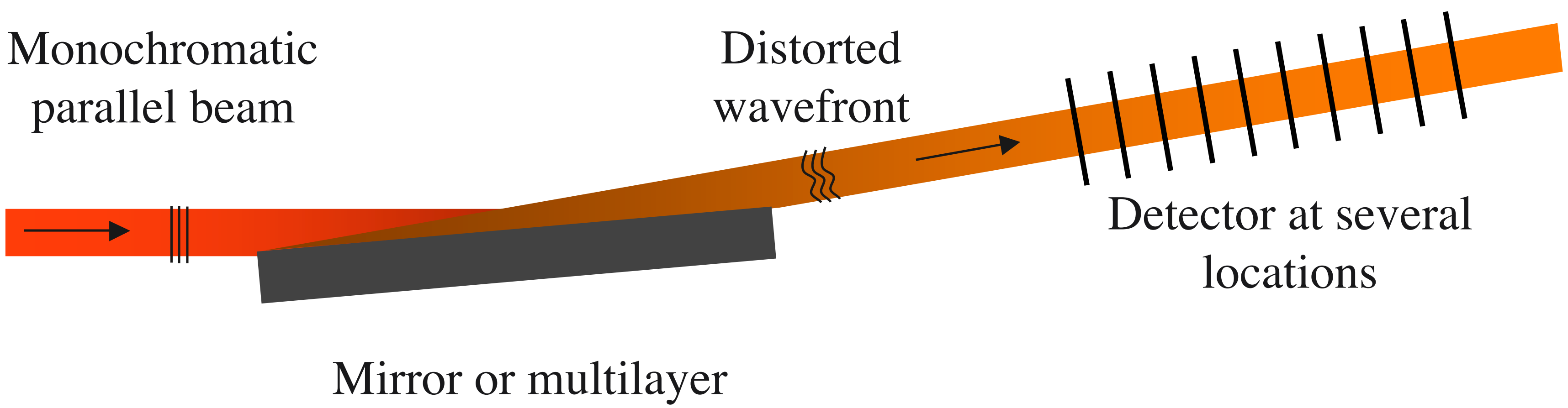
Abstract

Silicon polished by means of chemical-mechanical-polishing has been studied. A finely creviced top surface was seen in the AFM data. The power-spectral-density was measured by means of interferometry and by atomic force microscopy, and a roughness value of 0.21-0.23 nm rms was found by integration over the spatial frequency range that corresponds to our x-ray reflectivity data. Reflectivity data for 10 keV x-rays were obtained at the Advanced Photon Source, and a roughness of 0.22 – 0.30 nm was found to be roughly consistent with these data. A surface layer with a slightly higher electron density than that of crystalline silicon was needed to model the x-ray reflectivity. Crevices 3.6 nm deep and resulting in land areas having 85% coverage were invoked for the modeling. A total layer thickness of 7.4 nm was invoked for the modeling. That is, the crevices penetrated roughly half way through the total layer thickness. Due to the overall agreement between the two very different techniques for measuring roughness, namely, PSD and x-ray reflectivity data, we consider these results to accurately quantify roughnesses for a silicon surface that is near the state-of-art for smoothness.

Submitted to J. of Appl. Phys.

At wavelength metrology: Phase retrieval

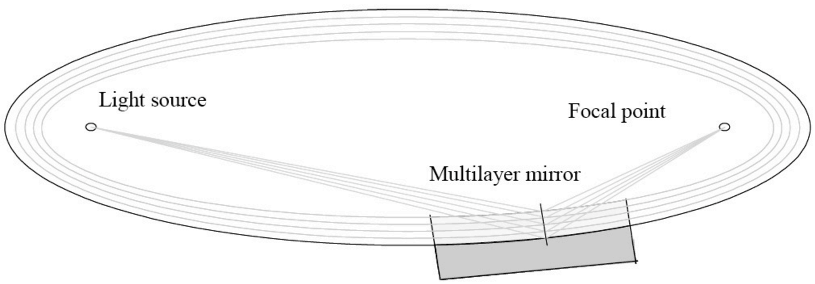
Phase retrieval from intensity distribution data taken at several distances from the mirror, can be used to reconstruct the mirror surface profile.

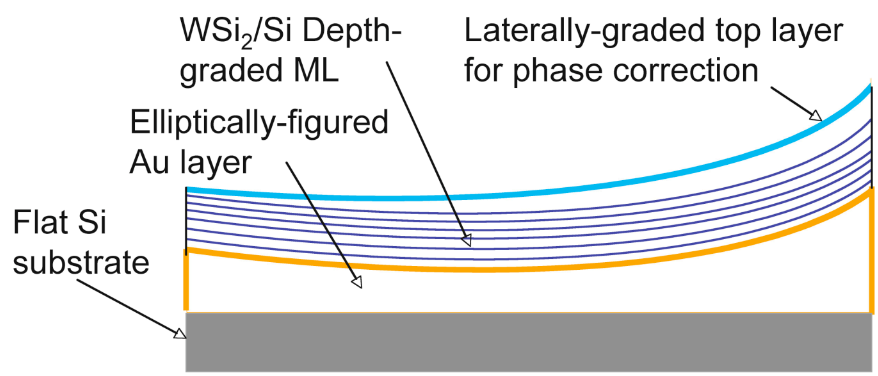


Metrology of graded KB ML for Hard X-ray Nanofocusing

Optics and Fabrication Group, Advanced Photon Source

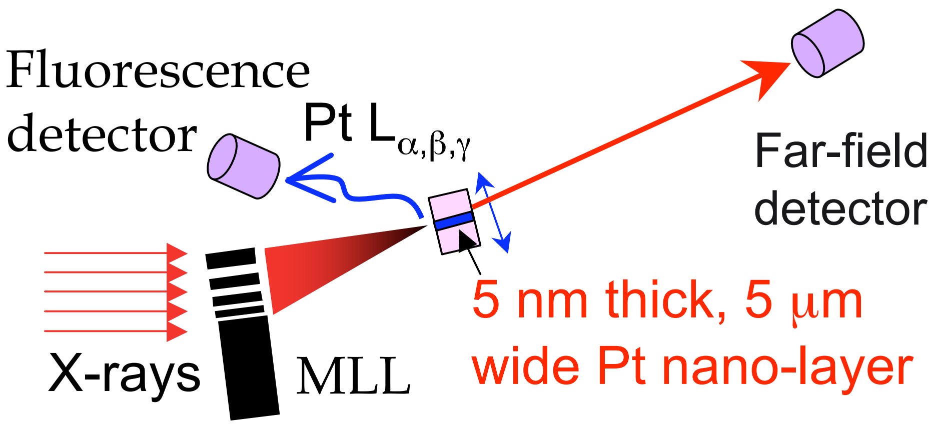
- The depth-graded ML profile ensures that the reflectivity is relatively constant over a wide angular range.
- However, the phase of complex reflectivity is perturbed, and the focus is aberrated.
- A laterally-graded surface layer on top of the ML stack introduces a path length difference to correct the phase aberration.



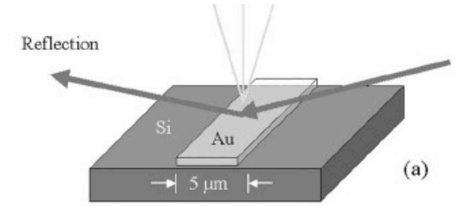


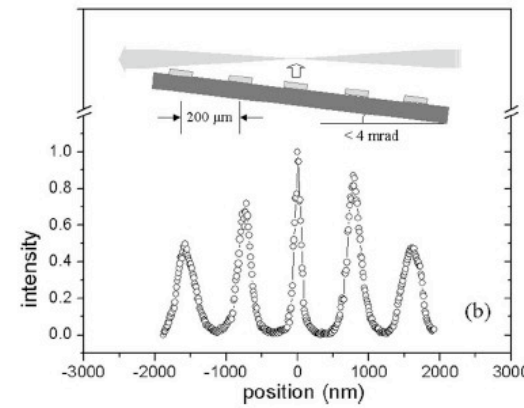
- Metrology for quality control and focus measurements:
  - Optical metrology not sufficient
  - At-wavelength metrology tools are being developed for both figure profile optimization and focal spot size measurements

At-wavelength metrology: techniques for focus measurements

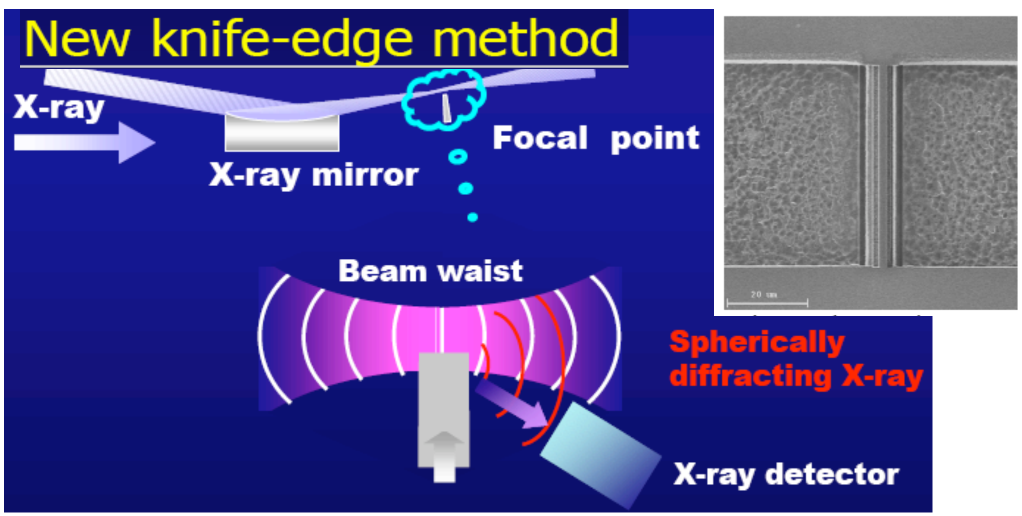


Fluorescence on a Pt nanowire: H-C. Kang, J. Maser, H. Yan, G.B. Stephenson, (APS/CNM nanoprobe, Argonne)

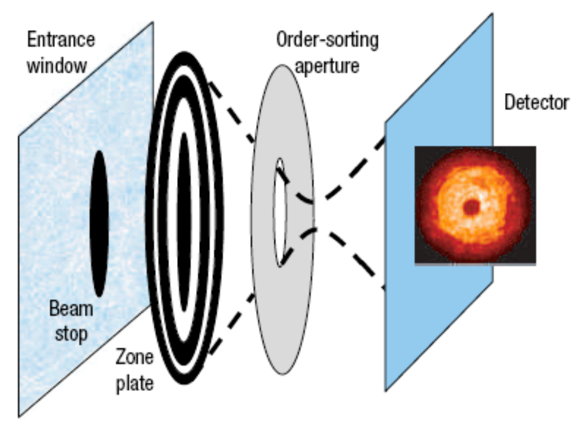




Fluorescence or reflection on a patterned Au mask : W. Liu *et al.* Rev. of Scien. Instrum. 76 (2005).



Mimura et al., SPIE Proc. (2007).



Phase Retrieval: H. M. QUINEY et al., Nature Physics Vol. 2 (2006).

Summary

- Future synchrotron radiation sources will provide beams with ultralow emittance and high brightness.
- Mirrors with slope errors (rms, standard deviation) in the nanoradian range and roughness <1 Å rms may be required to preserve the beam quality of these sources.
- This presents major challenges for both fabrication and metrology.
- Both optical and x-ray metrology tools are to be developed to overcome these challenges.
- At-wavelenght/in-situ metrology may become essential for optics development, particularly for coherence-preserving optics, ultraprecise ML structures, and nanofocusing optics. A number of x-ray wavefront and metrology techniques are being developed worldwide.
- A dedicated metrology beamline at a synchrotron radiation facility in the USA would be extremely useful not only for optics development but also for the development, calibration, and validation of ultra-precise optical metrology instruments and techniques.
- NLSL-II with its ultralow emittance and high brightness would be ideal for a metrology beamline.

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